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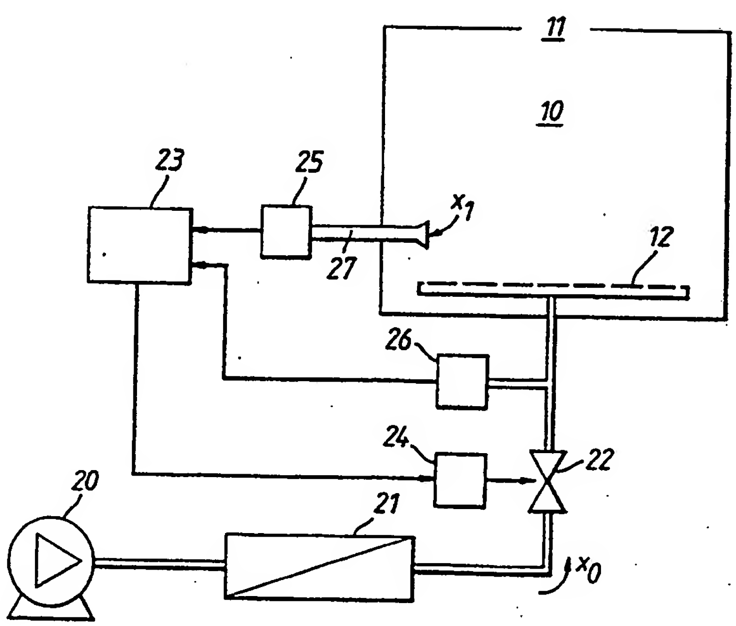

(57) Abstract

A method of purging a critical component such as oxygen from a container is described by feeding purging gas into the container until the concentration of oxygen falls to a target level. The level of oxygen in the purging gas is reduced during purging in response to the concentration of oxygen in the container. The purging gas is applied via a gas separation device, the output composition of which is dependant on the gas flow rate therethrough and the concentration of oxygen in the purging gas is varied during purging by varying the gas flow rate through the gas separation device. So long as the level of oxygen in the container exceeds the target level, the level of oxygen in the purging gas is lower than in the container, and, during the initial portion of the purging process, is higher than the target value.

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METHOD AND APPARATUS FOR PURGING A CONTAINER

The present invention relates to systems for purging a critical component from containers and the like with gas, generally an inert gas such as nitrogen.

A method of purging containers of a critical component such as oxygen is known, in which a purging gas is fed into the container and usually gas containing the critical component is allowed to exit from the container, until the concentration of the critical component falls to a target level. There are many situations in which it is desirable to purge a container which initially contains a normal oxygen-containing atmosphere, by replacing that normal atmosphere by one with a low oxygen concentration. One class of examples includes tanks which contain flammable fluids such as residual hydrocarbons or other oxidizable materials; with these, it is necessary to reduce the oxygen concentration to about 5%. Another class of examples is containers for grains and other foodstuffs which may be infested with insects or other pests. These can be eliminated by purging with nitrogen to reduce the oxygen level to about 1% to 2% for a controlled period. Such treatment has been shown to be an effective alternative to the use of chemical toxins, which can leave harmful residues in or on the foodstuff.

Any convenient source of nitrogen can be used for such purging. Liquid nitrogen and cylinders containing nitrogen at high pressure are long-established sources. The pressure swing absorption technique, using materials which differentially absorb one of the components (generally oxygen) from air under pressure and then release it when the pressure is reduced, has also become established for certain

situations. Recently, membrane air separation systems, in which a membrane is used which is differentially permeable to different components of air, have also come into commercial use for some situations, including purging.

In selecting a nitrogen source for purging, a variety of factors such as the initial cost of the system, the running cost, the size and weight of the system, convenience of use, and the time taken for the purging all have to be considered. For many situations, the use of liquid or pressurized nitrogen is ruled out by the complexities of organising regular supplies. The use of a pressure swing absorption or a membrane air separation system is therefore indicated in many situations. Of these two systems, the latter is often preferable, as it is considerably simpler than a pressure swing absorption system (which involves the complications of cycling between two states, and often of using at least two vessels as well).

With both the pressure swing absorption system and the membrane air separation, the bulk and capital cost of the system can be substantial, and will generally increase broadly proportionally as the desired flow rate of the purging gas is increased. In many situations, however, the purging rate must not be too slow, for cost, convenience, and/or other performance reasons.

It is an object of the present invention to maximise the purging rate achievable with a purging gas system of given size or other physical characteristics.

We have discovered that this objective can be achieved by controlling the composition of the purging gas during

purging, in response to the concentration of the critical component in the container.

Thus, according to a first aspect of the invention there is provided a method of purging a critical component from a container by feeding purging gas into the container until the concentration of the critical component falls to a target level, characterised in that the level of the critical component in the purging gas is varied during purging in response to the concentration of the critical component in the container.

According to a second aspect of the invention, there is provided an apparatus for purging a critical component from a container to a target level, comprising: i) means for feeding purging gas to the container; ii) means for measuring the concentration of the critical component in the container; and iii) control means for varying the concentration of the critical component in the purging gas in response to the measured concentration of the critical component in the container.

The apparatus may further comprise: iv) means for measuring the concentration of the critical component in the purging gas, said control means additionally being responsive to the measured concentration of the critical component in the purging gas.

Usually the critical component is oxygen although the method and apparatus according to the invention are equally applicable to the purging of other components, especially other gaseous components from containers. The purging gas will usually comprise, in addition to the critical component, an inert component such as nitrogen. Throughout

the following description, the critical component referred to is oxygen, but it is to be understood that the description applies equally to other critical components.

We prefer that, in a first phase of the purging process, the level of oxygen in the purging gas is lower than the level of oxygen in the container, so long as the level of oxygen in the container exceeds the target level and ideally that, during at least a portion of the purging process, the level of oxygen in the purging gas is higher than the target value. This can be achieved in a preferred embodiment of the invention, when the concentration of oxygen in the purging gas is reduced during purging. Once the level of oxygen in the purging gas falls to the target value, the second phase of the process is reached in which the level of oxygen in the purging gas is set at or below the target value. Once the level of oxygen in the container reaches the target value, the level of oxygen in the purging gas may then be maintained at a level such as to maintain the target level in the container. In an ideal situation, this will mean maintaining the oxygen level of the purging gas at the target value. However, in a situation where leakage into the container occurs, for example leakage of air, it may be necessary to maintain the level of oxygen in the purging gas below the target value.

Thus, the control means may be programmed to set the concentration of oxygen in the purging gas (x_o) in response to the concentration of oxygen in the container (x_1) and the target level (x_t) according to the formula

$$x_o = (x_1 - a),$$

so long as

$$x_o > x_t$$

where "a" has a positive value. Usually, the value of "a" is a constant during at least one portion of the purging process.

In the second phase where the concentration of oxygen in the container (x_1) still lies above the target level (x_t), the control means may be programmed to set the concentration of oxygen in the purging gas (x_o) in response to the concentration of oxygen in the container (x_1), according to the formula

$$(x_1 - a) \leq x_o \leq x_t.$$

In the third phase, the control means may be programmed to set the concentration of oxygen in the purging gas (x_o) in response to the concentration of oxygen in the container (x_1) and the target level (x_t) according to the formula

$$x_o \leq x_t,$$

as soon as

$$x_1 = x_t.$$

In more sophisticated embodiments, the concentration of oxygen in the purging gas (x_o) may be set in response to the rate of change of the concentration of oxygen in the container (x_1).

The means for feeding purging gas to the container preferably comprises a gas separation device, such as a membrane separator, the output composition of which is dependant on the gas flow rate therethrough. The means for varying the concentration of the purging gas may then comprise means for varying the flow rate of gas through the membrane separator. Thus, the concentration of oxygen in the purging gas is then be varied during purging by varying the gas flow rate through the gas separation device. It is a characteristic of certain purging gas systems (including in particular membrane air separation systems) that the gas flow rate and the composition of the output gas can readily be controlled, with the gas flow rate being approximately inversely related to the output gas composition (or more precisely the nitrogen content of the output gas). In such systems, the application of the invention enables the time required to achieve purging to be decreased by suitably controlling these variables during purging, with the purge gas flow rate being initially high and the nitrogen concentration being below that required, and the purge gas flow rate being reduced and the nitrogen concentration being increased as purging proceeds.

The invention is particularly advantageous where the container has an internal structure or arrangement of void space which allows significant mixing of the injected purge gas with the atmosphere being displaced, e.g. air.

While not wishing to be bound by theory, it is believed that the benefit of the invention can be explained as follows, in the case where the critical gas is oxygen and is supplied by a gas separator or the like, the output concentration of which is a function of the gas flow rate therethrough:

If purge gas is fed in at the target oxygen concentration from the beginning, this produces a high oxygen concentration gradient in the container. This results in a high diffusion flow of oxygen, which is equivalent to a high degree of mixing between the original normal or near normal air and the low oxygen purge gas. According to the invention the oxygen concentration gradient is kept low, so that the degree of mixing is low and the effect just noted is minimized. In addition, the purge gas flow rate is initially considerably higher (until the oxygen concentration approaches the target level), so that the rate of displacement of the normal or near normal air is correspondingly increased. The overall result is that the purge time (i.e. the time to reach the target oxygen concentration substantially throughout the container) is considerably reduced, as noted above.

The high initial purge gas flow rate of the present system has another important advantage. In some large containers, it is possible for inversion to occur, resulting eg from heating of parts of the container by sunlight, with undesirable results (eg mass flow of the normal or near-normal air to the bottom of the container and of the low oxygen gas to the top of the container and its subsequent displacement out of the container). The chance of such inversion effects is considerably reduced if the flow rate of the purging gas is relatively high.

A purge system embodying the invention will now be described, by way of example, with reference to the drawings, in which:

Fig 1 is a block diagram of the purge system applied to a container; and

Fig 2 is a graph showing the operation of the purge system.

Referring to Fig. 1, a container 10 has a vent 11 at its top and a purge gas inlet system 12 at its bottom. The container may contain some form of goods (not shown). A compressor 20 supplies air under pressure to a membrane air separation unit 21, in which oxygen diffuses away differentially, producing a nitrogen-enriched output gas, which is fed through a needle valve 22 to the purge gas inlet system 12 at the bottom of the container 10.

To implement the strategy of the present invention, the valve 22 is controlled to allow a high initial purge gas flow rate and then to progressively reduce the flow rate until a target concentration of nitrogen is achieved over a substantial proportion of the container.
The valve 22 is controlled by a control unit 23 via an actuator 24. The system includes two oxygen concentration sensors, 25 and 26, the first of which senses the oxygen concentration at a suitable point in the container 10 by means of an intake 27 and the second of which senses the oxygen concentration in the purge gas from the valve 22. These two sensors feed the controller 23, which adjusts the purge gas flow rate in dependence on their outputs. The controller should have a high proportional band, preferably in excess of 500%, in order to avoid "hunting" of the control point in the air separator flow rate (and injected oxygen level). The ideal proportional band will depend <u>inter alia</u> on the position of the intake 27 and the volume of the container. The further this intake is from the inlet system 12, and/or the larger the volume of the container, the higher will be the ideal proportional band for the controller. A suitable such controller is a West 2075

Controller ex West Instruments, Brighton, UK having a proportional band of the order of 1000%.

It is possible to take a number of samples from the container by, for example, arranging for two or more intakes 27 to be provided at the same horizontal level, and mixing the sampled gas before analysis or by averaging the analysis results. This helps to overcome the effects of the intake 27 being positioned in a dead space within the container.

The controller can conveniently operate as follows. For as long as the purge gas oxygen content is greater than the target oxygen content, the purge gas flow rate is adjusted to maintain the oxygen content of the purge gas at a fixed level below the oxygen content in the container (at the sensor); then, when the oxygen content in the container equals the target value, the purge gas oxygen content is held at the container (and target) oxygen level. (The pressures involved are all small compared with atmospheric, so the oxygen concentrations can be taken indifferently as absolute or relative.)

These operating conditions can be expressed symbolically as follows. Take

x_o = the oxygen concentration in the injected purge gas (as sensed by sensor 26)

x_1 = the oxygen concentration in the gas in the container (as sensed by sensor 25)

x_t = the target oxygen concentration.

a = a constant.

Then

$$\text{IF } x_o > x_t \text{ THEN SET } x_o = (x_1 - a) \qquad (1)$$

$$\text{IF } x_1 = x_t \text{ THEN SET } x_o = x_t \qquad (2)$$

An example of the operation of this system is shown in Fig. 2, which shows the purge gas oxygen content (PG O_2%) against time. The atmospheric oxygen content of 21% O_2 (+ 70% N_2) is shown as line 30. The purge gas oxygen content starts off at a high value (about 19%), which is reduced along a curve 31 during which $x_o > x_t$ (i.e. condition (1) above applies). At about time t = 24, this condition ceases to apply, and $x_o = x_t$ (i.e. condition (2) above applies), and from that time onwards, the oxygen content of the purge gas is kept constant at 5% (curve 32). In this example, the oxygen content of the container was reduced to 10% at time t = 80 (point 33). In contrast, the simple known strategy of having the oxygen content of the purge gas held steady at 5% for the whole time (curve 34) reduced the oxygen content of the container only as far as 18% by time t = 80 (point 35). After a further period of time, not shown in Figure 2, the oxygen content of the container x_1 reaches the target value of 5%. Thereafter, the purging gas oxygen content is maintained at 5% in order to maintain the container gas oxygen concentration at that level.

The test intake 27 is preferably located fairly low in the container 10, but far enough above the base of the container to make the effects of minor variations of the oxygen concentration insignificant and to allow reasonably accurate sensing of the oxygen gradient. The constant "a" is chosen to give an oxygen gradient which results in relatively high purge gas flow rates and relatively low diffusion mixing in the container.

Depending on the nature and purpose of the purge, the purge system can be kept running either permanently (for as long as the purge is to be maintained) or for a fixed time thereafter (sufficient as determined eg by experience to establish the target oxygen concentration throughout the container).

The oxygen content of the purge gas can of course be controlled in various ways. The precise control equations used, i.e. the precise curve which the oxygen content is caused to follow with time, can of course have more than two segments, and each segment can be straight or curved according to any convenient formula (e.g. quadratic or exponential). Also, the time lengths of the segments and the parameters of the curve in those segments can be controlled in various ways. In the system above, the oxygen concentrations in the purge gas and the container are sensed, but other variables (eg the purge gas flow rate and/or the quantity of material in the container) can be sensed and used for control, or the curve may be predetermined.

CLAIMS

1. A method of purging a critical component from a container by feeding purging gas into the container until the concentration of the critical component falls to a target level, characterised in that the level of the critical component in the purging gas is varied during purging in response to the concentration of the critical component in the container.

2. A method according to claim 1, wherein the concentration of the critical component in the purging gas is reduced during purging.

3. A method according to claim 1 or 2, wherein the purging gas is supplied via a gas separation device, the output composition of which is dependant on the gas flow rate therethrough.

4. A method according to claim 3, in which the concentration of the critical component in the purging gas is varied during purging by varying the gas flow rate through the gas separation device.

5. A method according to any preceding claim, wherein the level of the critical component in the purging gas is lower than the level of the critical component in the container, so long as the level of the critical component in the container exceeds the target level.

6. A method according to any preceding claim, wherein, during at least a portion of the purging process, the level of the critical component in the purging gas is higher than the target value.

7. A method according to any preceding claim, wherein the critical component is oxygen.

8. An apparatus for purging a critical component from a container to a target level, comprising:

i) means for feeding purging gas to the container;

ii) means for measuring the concentration of the critical component in the container; and

iii) control means for varying the concentration of the critical component in the purging gas in response to the measured concentration of the critical component in the container.

9. An apparatus according to claim 8, further comprising:

iv) means for measuring the concentration of the critical component in the purging gas, said control means additionally being responsive to the measured concentration of the critical component in the purging gas.

10. An apparatus according to claim 9, wherein said control means is programmed to set the concentration of the critical component in the purging gas (x_o) in response to the concentration of the critical component in the container (x_1) and the target level (x_t) according to the formula

$$x_o = (x_1 - a),$$

so long as

$$x_o > x_t$$

where "a" has a positive value.

11. An apparatus according to claim 10, wherein said control means is programmed to set the concentration of the critical component in the purging gas (x_o) in response to the concentration of the critical component in the container (x_1) and the target level (x_t) according to the formula

$$(x_1 - a) \leq x_o \leq x_t,$$

so long as

$$x_1 > x_t.$$

12. An apparatus according to claim 10 or 11, wherein said control means is programmed to set the concentration of the critical component in the purging gas (x_o) in response to the concentration of the critical component in the container (x_1) and the target level (x_t) according to the formula

$$x_o \leq x_t,$$

as soon as

$$x_1 = x_t.$$

13. An apparatus according to claim 10 or 11, wherein the value of "a" is a constant during at least one portion of the purging process.

14. An apparatus according to any one of claims 8 to 13, wherein the means for supplying purging gas to the container includes a membrane separator.

15. An apparatus according to claim 14, wherein the means for varying the concentration of the purging gas comprises means for varying the flow rate of gas through the membrane separator.

AMENDED CLAIMS

[received by the International Bureau on 29 July 1993 (29.07.93); original claim 1 amended; remaining claims unchanged (1 page)]

1. A method of reducing infestation in a container by purging a critical component therefrom by feeding purging gas into the container until the concentration of the critical component falls to a target level, characterised in that the level of the critical component in the purging gas is varied during purging in response to the concentration of the critical component in the container.

2. A method according to claim 1, wherein the concentration of the critical component in the purging gas is reduced during purging.

3. A method according to claim 1 or 2, wherein the purging gas is supplied via a gas separation device, the output composition of which is dependant on the gas flow rate therethrough.

4. A method according to claim 3, in which the concentration of the critical component in the purging gas is varied during purging by varying the gas flow rate through the gas separation device.

5. A method according to any preceding claim, wherein the level of the critical component in the purging gas is lower than the level of the critical component in the container, so long as the level of the critical component in the container exceeds the target level.

6. A method according to any preceding claim, wherein, during at least a portion of the purging process, the level of the critical component in the purging gas is higher than the target value.

STATEMENT UNDER ARTICLE 19

Claim 1 of the application has been amended to direct the invention to a method of reducing infestation in a container. A basis for this amendment can be found on page 1, second paragraph of the application as filed:

> "Another class of examples is containers for grains and other foodstuffs which may be infested with insects or other pests."

The International Search Report refers to WO91/11913 (The Broken Hill Proprietary Company Limited) which is concerned with the generation of controlled atmospheres for use in horticultural applications. Broken Hill makes no reference to the reduction of infestation in a container. The Broken Hill document is concerned only with respiring horticultural produce, such as fruit and vegetables. The present application is concerned with containers for foodstuffs such as grains, which are non-respiring. Broken Hill assumes perfect mixing in the container (see page 7, line 2), whereas the present invention is based on reducing the degree of mixing in the container - see page 7, first paragraph of the present application:

> "According to the invention the oxygen concentration gradient is kept low, so that the degree of mixing is low....."

The Applicants submit therefore that their invention is both novel and involves an inventive step over the disclosures of Broken Hill.

The amendment to Claim 1 will necessitate corresponding amendment to the description, on page 3, paragraph 1.

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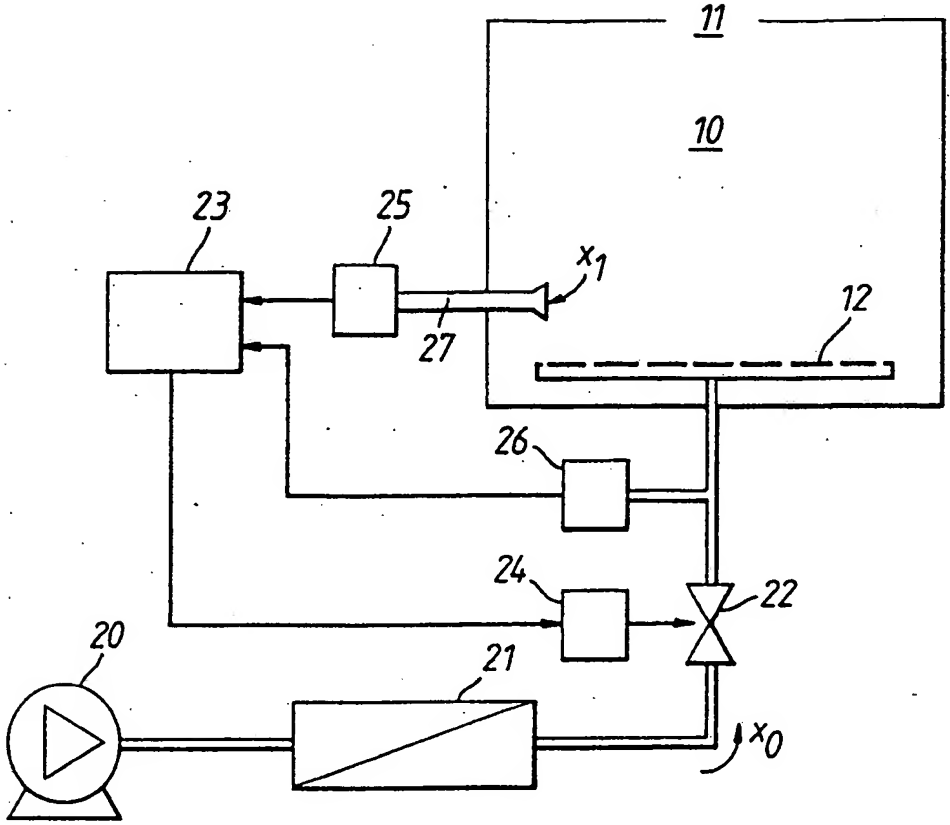
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Fig.1.

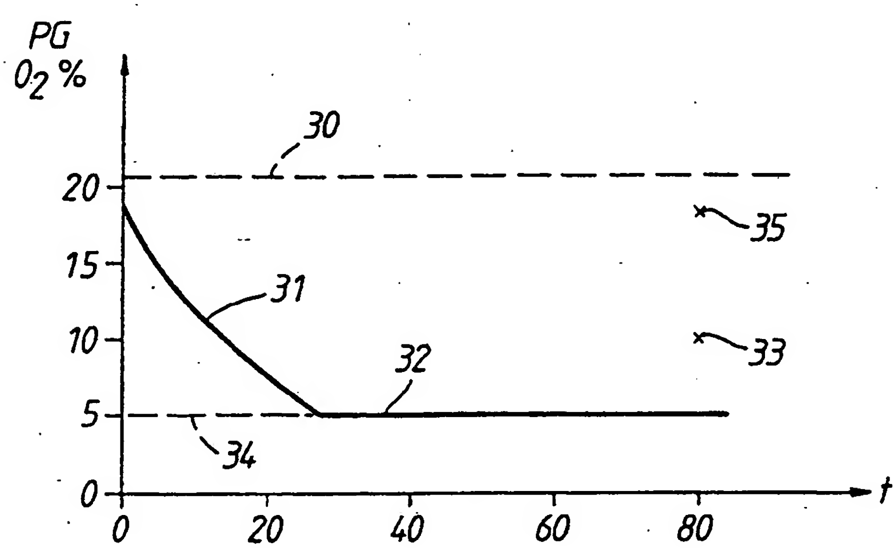
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Fig.2.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/GB 93/00428

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: B65D 90/44

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: A23B, A23L, B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO, A1, 9111913 (THE BROKEN HILL PROPRIETARY COMPANY LIMITED), 22 August 1991 (22.08.91), page 9, line 34 - page 10, line 16, claims 4-10	1-15
	--	
A	US, A, 4317797 (SMITH), 2 March 1982 (02.03.82)	1-15
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☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
14 May 1993	

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT
Information on patent family members

31/03/93

International application No.
PCT/GB 93/00428

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO-A1- 9111913	22/08/91	NONE	
US-A- 4317797	02/03/82	NONE	

Form PCT/ISA/210 (patent family annex) (July 1992)